

## PATENT ABSTRACTS OF JAPAN

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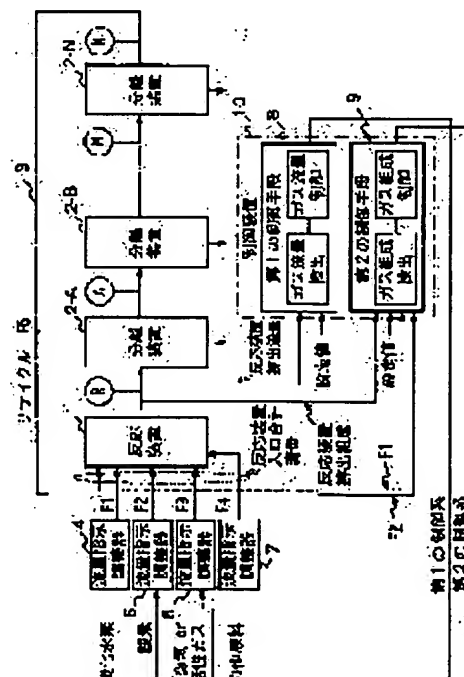
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## (54) GAS PHASE OXIDATION REACTION SYSTEM AND METHOD AND DEVICE FOR CONTROLLING THE SAME

(57)Abstract:

**PROBLEM TO BE SOLVED:** To provide a method and device for efficiently and stably operating a gas phase oxidation reaction system for hydrocarbons having a process to recover unreacted hydrocarbon and circulate in a reaction vessel.

**SOLUTION:** In a gas phase oxidation reaction system equipped with at least one reaction apparatus in which a raw material containing hydrocarbons, oxygen and air or an inactive gas is fed, one or more of separators connected to the reaction apparatus and a recycle loop for returning a discharged flow from either one or more of the separators to the reaction apparatus as a part of the feed flow, the gas phase oxidation reaction system is characterized in that a first controlling system for controlling one or both of the sum total flow at the inlet of the



reaction apparatus or the discharged flow from the reaction apparatus and the discharged flow from one or more of stages of the aforesaid one or more of separators and a second controlling system for controlling the composition of the whole feed flow to the reaction apparatus or the gas composition at the outlet of the reaction apparatus or the outlet at either one or more of the outlets of the separators are installed to be parallel.

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## LEGAL STATUS

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**CLAIMS**

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**[Claim(s)]**

[Claim 1] At least one reactor with which the raw material containing a hydrocarbon, oxygen and air, or inert gas is supplied, It is the vapor-phase-oxidation reaction system equipped with the recycle loop formation which returns one combined with this reactor or the decollator beyond it, and the outflow from any one or more decollators to a reactor as a part of feeder current. The sum total flow rate of a reactor inlet port or the amount of outflow from this reactor, and the 1st control system by which even the above controls one side or the both sides of each stage of a decollator with the amount of outflow from any one or more to a request value, [ beyond it ] The vapor-phase-oxidation reaction system characterized by establishing in parallel the 2nd control system which controls one side or the both sides of a gas presentation of a presentation or reactor outlet of all the feeder current to a reactor, or the outlet of any one or more decollators to a request value.

[Claim 2] At least one reactor with which the raw material containing a hydrocarbon, oxygen and air, or inert gas is supplied, It is the control approach of the vapor-phase-oxidation reaction system equipped with the recycle loop formation which returns one combined with this reactor or the decollator beyond it, and the outflow from any one or more decollators to a reactor as a part of feeder current. As the 1st control, the sum total flow rate or the amount of outflow from this reactor of a reactor inlet port, Even the above or one side or the both sides from any one or more of each stage of a decollator with the amount of outflow beyond it It controls to a request value by changing the flow rate of the air supplied to a vapor-phase-oxidation reaction system, or inert gas. Further as the 2nd control Even the above the oxygen density in one side or the both sides of each stage of a decollator with outflow from either with the outflow from a reactor [ beyond it ] The control approach of the vapor-phase-oxidation reaction system characterized by what is controlled to a request value by changing the flow rate of the hydrocarbon supplied to a vapor-phase-oxidation reaction system, or the flow rate of oxygen based on the flow rate of the hydrocarbon supplied to a vapor-phase-oxidation reaction system, and oxygen.

[Claim 3] At least one reactor with which the raw material containing a hydrocarbon, oxygen and air, or inert gas is supplied, It is the control unit of the vapor-phase-oxidation reaction system equipped with the recycle loop formation which returns one combined with this reactor or the decollator beyond it, and the outflow from any one or more decollators to a reactor as a part of feeder current. The sum total flow rate of a reactor inlet port or the amount of outflow from this reactor, and even the above or one side or the both sides from any one or more of each stage of a decollator with the amount of outflow beyond it The 1st control means controlled to a request value by changing the flow rate of the air supplied to a vapor-phase-oxidation reaction system, or inert gas, Even the above the oxygen density in one side or the both sides of each stage of a decollator with outflow from either with the outflow from a reactor [ beyond it ] The control unit of the vapor-phase-oxidation reaction system characterized by having the 2nd control means controlled to a request value by changing the flow rate of the hydrocarbon supplied to a vapor-phase-oxidation reaction system, or the flow rate of oxygen based on the flow rate of the hydrocarbon supplied to a vapor-phase-oxidation reaction system, and oxygen.

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**DETAILED DESCRIPTION**

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**[Detailed Description of the Invention]****[0001]**

**[Field of the Invention]** This invention offers the control technique which enables operation of the vapor-phase-oxidation reaction system to which a hydrocarbon and oxygen content gas are made to react under existence of a catalyst to stability about the control approach of a vapor-phase-oxidation reaction system, and a control unit.

**[0002]**

**[Description of the Prior Art]** Under existence of a catalyst, as a vapor-phase-oxidation reaction to which a hydrocarbon and oxygen content gas are made to react, there is the catalytic-oxidation approach of a hydrocarbon, for example, and manufacture of the maleic anhydride by oxidation of the hydrocarbon of a carbon number 4, manufacture of the acrylonitrile by the ammoxidation of the hydrocarbon of a carbon number 3, manufacture of the ethylene dichloride by the oxychlorination of ethylene, etc. are known widely. In recent years, in order to produce the target product efficiently in these vapor-phase-oxidation reactions, while raising the selectivity to a product by stopping the invert ratio of the hydrocarbon in a reactor low, the recycling law through which collects unreacted hydrocarbons and a reactor is made to circulate is proposed.

**[0003]** However, although operation was able to be continued to stability in the conventional process by controlling a reaction condition (the flow rate of each gas, and temperature, a pressure, the amount of catalysts) independently, respectively when reaction results changed, for example In the case of a recycle process, in order that the gas which came out of the reactor may finally return to a reactor via a decollator etc. again For example, when reaction results change, even if it was going to perform a reaction conditional control like before, in order that the flow rate of recycle gas and a presentation might change influenced by the reaction condition of modification, if it remained as it is, it was difficult [ it ] to fall and wear in the regular condition, and for there to be nothing and to continue oxidation reaction to stability as a result with Lycium chinense.

**[0004]**

**[Problem(s) to be Solved by the Invention]** This invention aims the vapor-phase-oxidation reaction system of the hydrocarbon which has the recycle process through which collects unreacted hydrocarbons and a reactor is made to circulate at efficient and offering the control approach for operating to stability, and a control unit.

**[0005]**

**[Means for Solving the Problem]** At least one reactor with which the raw material with which this invention contains a hydrocarbon, oxygen and air, or inert gas is supplied, In the vapor-phase-oxidation reaction system equipped with the recycle loop formation which returns one combined with this reactor or the decollator beyond it, and the outflow from any one or more decollators to a reactor as a part of feeder current By establishing in parallel the 1st control system which controls directly or indirectly the sum total flow rate of a reactor inlet port, and the 2nd control system which controls the distributed gas presentation to a reactor, or the exhaust-gas presentation from a reactor to a request value Efficient and stable operation of a system is enabled and it is constituted as follows.

**[0006]** (1) The vapor-phase-oxidation reaction system by this invention At least one reactor with which the raw material containing a hydrocarbon, oxygen and air, or inert gas is supplied, It has the recycle loop formation which returns one combined with this reactor or the decollator beyond it, and the outflow from any one or more decollators to a reactor as a part of feeder current. The sum total flow rate of a reactor inlet port or the amount of outflow from this reactor, and the 1st control system by which even the above controls one side or the both sides of each stage of a decollator with the amount of outflow from any one or more to a request value, [ beyond it ] It is characterized by the configuration which established in parallel the 2nd control system which controls one side or the both sides of a gas presentation of the presentation of all the feeder current to a reactor, the outlet of a reactor, or the outlet of any one or more decollators to a

request value.

[0007] (2) The control approach of the vapor-phase-oxidation reaction system by this invention At least one reactor with which the raw material containing a hydrocarbon, oxygen and air, or inert gas is supplied, In the vapor-phase-oxidation reaction system equipped with the recycle loop formation which returns one combined with this reactor or the decollator beyond it, and the outflow from any one or more decollators to a reactor as a part of feeder current As the 1st control, the sum total flow rate or the amount of outflow from this reactor of a reactor inlet port, Even the above or one side or the both sides from any one or more of each stage of a decollator with the amount of outflow beyond it It controls to a request value by changing the flow rate of the air supplied to a vapor-phase-oxidation reaction system, or inert gas. Further as the 2nd control Even the above the oxygen density in one side or the both sides of each stage of a decollator with outflow from either with the outflow from a reactor [ beyond it ] It is characterized by the configuration controlled to a request value by changing the flow rate of the hydrocarbon supplied to a vapor-phase-oxidation reaction system, or the flow rate of oxygen based on the flow rate of the hydrocarbon supplied to a vapor-phase-oxidation reaction system, and oxygen.

[0008] (3) The control unit of the vapor-phase-oxidation reaction system by this invention At least one reactor with which the raw material containing a hydrocarbon, oxygen and air, or inert gas is supplied, It has the recycle loop formation which returns one combined with this reactor or the decollator beyond it, and the outflow from any one or more decollators to a reactor as a part of feeder current. The sum total flow rate of a reactor inlet port or the amount of outflow from this reactor, and even the above or one side or the both sides from any one or more of each stage of a decollator with the amount of outflow beyond it The 1st control means controlled to a request value by changing the flow rate of the air supplied to a vapor-phase-oxidation reaction system, or inert gas, Even the above the oxygen density in one side or the both sides of each stage of a decollator with outflow from either with the outflow from a reactor [ beyond it ] It is characterized by the configuration which has the 2nd control means controlled to a request value by changing the flow rate of the hydrocarbon supplied to a vapor-phase-oxidation reaction system, or the flow rate of oxygen based on the flow rate of the hydrocarbon supplied to a vapor-phase-oxidation reaction system, and oxygen.

[0009] The outline configuration of the vapor-phase-oxidation reaction system which applied this invention is shown in drawing 1 by the instantiation-approach. Hereafter, the detail of this invention is explained based on drawing 1. The vapor-phase-oxidation reaction system of drawing 1 has at least one reactor 1, one, or decollator 2-A beyond it - 2-N, and recycle loop formation 3. A part for new supply of the hydrocarbon supplied to a reactor 1, oxygen, air or inert gas, and other gaseous-phase raw materials is adjusted to a flow rate F1, F2, F3, and F4 by the flow rate directions regulators 4, 5, 6, and 7, respectively. Moreover, although the illustration abbreviation is carried out, the catalyst is made to exist in a reactor 1.

[0010] The resultant from a reactor 1, a by-product, or an unnecessary component is taken out from each of decollator 2-A - 2-N by arbitration, or is removed. R points show the outflow from a reactor 1, and, as for an A point, the outflow from first decollator 2-A, --, N point show the outflow from Nth decollator 2-N. The outflow from last or Nth decollator 2-N is recycled by the reactor 1 through the recycle loop formation 3 as a part of feeder current. This recycle flow rate is expressed with F5. Thereby, the sum total flow rate of the inlet port of a reactor 1 is given by  $(F1+F2+F3+F4+F5)$ . Moreover, the presentation of all the feeder current to a reactor 1 is given with the sum total flow rate  $(F1+F2+F3+F4+F5)$  of each component flow rate / reactor inlet port of a reactor inlet port.

[0011] Furthermore by this invention, at least two independent control systems, the 1st and the 2nd, are established respectively through the 1st control means 8 and 2nd control means 9. The 1st control system using the 1st control means 8 The sum total flow rate or the amount of outflow from this reactor of the inlet port of a reactor 1, It is for even the above to control one side or the both sides of each stage of a decollator with the amount of outflow from any one or more to a request value. In the example of illustration [ beyond it ] The flow rate of the outflow from a reactor 1 was detected by R points, and the flow rate F3 of the air supplied to a reactor 1 or inert gas is changed so that the detected flow rate may be in agreement with desired value. The 2nd control system Moreover, the presentation of all the feeder current to a reactor 1 or the outlet of a reactor 1, It is for controlling the gas presentation in the outlet of one or more things of decollator 2-A - 2-N to a request value. Or in the example of illustration The gas presentation of the outflow from a reactor 1 was detected by R points, and the flow rate F1/F2 of the hydrocarbon supplied to a reactor 1 and oxygen is changed so that the detected gas presentation may be in agreement with the set point. The material gas containing a hydrocarbon, oxygen, air, or inert gas may be directly supplied to a reactor or a decollator, and may be supplied to the feeder current or the outflow to a decollator.

[0012] As the 1st control means 8 and 2nd control means 9, it can use also with what type of PID (proportional-integral-derivative) control device also with what kind of a feedback mold control device or what kind of kind of feedforward mold control device. In the example shown in drawing 2, a control system including the 1st control means 8 controls the

flow rate directions regulator 6 to lower the air in a reactor inlet port, or the supply flow rate F3 of inert gas, when the amount of outflow measured by R points of for example, a reactor outlet increases to desired value, and it functions as maintaining the amount of outflow in a reactor outlet at a request value.

[0013] moreover, when the 1st control means 8 detects the flow rate of either the A point of each decollator outlet - N point, for example, the outflow of M point, unlike the example of illustration, the flow rate directions regulator 6 is controlled and the flow rate F3 of air or inert gas is changed so that the flow rate of M point may be maintained at a request value. Therefore, when the flow rate of M points decreases to the set point, it controls in the direction to which the flow rate F3 of air or inert gas is made to increase, and it operates so that the flow rate of M points may be maintained at a request value.

[0014] Next, the 2nd control means 9 measures the oxygen density in the outflow of R points, and a control system including the 2nd control means 9 controls it to lower the desired value of the flow rate (for example, it considers as an oxygen flow rate / hydrocarbon flow rate) of the hydrocarbon in a reactor inlet port, and oxygen, when the measured value increases to the desired value of the oxygen density set up beforehand. The control system of the flow rate of the hydrocarbon contained in the 2nd control means and oxygen measures the flow rate (for example, it considers as an oxygen flow rate / hydrocarbon flow rate) of the hydrocarbon of a reactor inlet port, and oxygen, when increasing to desired value, control the flow rate directions regulator 5 of the oxygen in a reactor inlet port, and the flow rate of the hydrocarbon of a reactor inlet port and oxygen is made to follow desired value, and it functions as keeping final the oxygen density in the outflow of R points to the desired value set up beforehand. Or the flow rate of the hydrocarbon of a reactor inlet port and oxygen is measured, when increasing to desired value, the flow rate directions regulator 4 of the hydrocarbon in a reactor inlet port may be controlled, the flow rate of the hydrocarbon of a reactor inlet port and oxygen may be made to follow desired value unlike the case of illustration, and, finally the oxygen density in the outflow of R points may be operated with maintaining at the desired value set up beforehand. In addition, when detecting and controlling the oxygen density in outflow about R points - two or more N points unlike the case of illustration, a control variable (valve flow coefficient) is constituted using the weighted average of each detection value, and other suitable performance indices, and it is made to maintain at desired value by the same control technique.

[0015] The exhaust-gas flow rate or recycle gas flow rate from each distributed gas flow rate, reactor, or decollator to a reaction system is measured by quantity-of-gas-flow measurement means usually used, such as a differential pressure type flowmeter, an eddy type flowmeter, a coriolis type flowmeter, and a variable area flowmeter. The oxygen density in the outflow from a reactor or a decollator is measured by oxygen density measurement means usually used, such as an analyzer which can measure various online oxygen analyzers and an oxygen density, and hydrocarbon concentration is also measured by various online analyzers, such as an infrared analyzer and a mass spectrometer. In addition, it is desirable to use the means which can measure [ rather than ] concentration continuously in this case using a discontinuous analysis means like a gas chromatograph.

[0016] A decollator may separate the resultant, by-product, or unnecessary component from a reactor or a decollator, and may divide the outflow from a reactor or a decollator into two or more flow. As a decollator, well-known decollators, such as an absorption decollator, a condensation decollator, adsorption separation equipment, and a distillation decollator, can usually be used.

[0017] This invention is applicable to manufacture of the acrylonitrile by the ammoxidation of the hydrocarbon of the carbon number 3 of manufacture of the maleic anhydride by oxidation of the hydrocarbon of the carbon number 4 of the vapor-phase-oxidation reaction to which a hydrocarbon and oxygen content gas are made to react, for example, butane, a butene, a butadiene, etc., a propane, a propylene, etc., and manufacture of the ethylene dichloride by the oxychlorination of ethylene under existence of a catalyst.

[0018] When manufacturing a maleic anhydride, nitrogen, a carbon dioxide, etc. are usually used for a raw material as the air to which the hydrocarbons of the carbon number 4 of butane, a butene, a butadiene, etc. were carried out as a hydrocarbon, and enrichment of air and the oxygen was carried out as oxygen content gas and oxygen or those mixed gas, and inert gas. Especially the thing that makes an active ingredient the multiple oxide (vanadium-Lynn system multiple oxide) which uses vanadium and Lynn as a main configuration element as a catalyst, and makes vanadic pyrophosphate an active ingredient especially is desirable.

[0019] Such a catalyst can be manufactured by the approach indicated by Chem.Rev.88, 55-80 pages (1988), JP,59-95933,A, the U.S. Pat. No. 4,472,527 specification, the U.S. Pat. No. 4,520,127 specification, etc. A reaction can use a fluid bed reactor or a fixed bed reactor, and is made to usually react at 300-600 degrees C.

[0020] In the reaction generation gas which flows out of a reactor, unreacted oxygen and coal-for-coke-making-ized hydrogen, the carbon monoxide which carries out a byproduction to a list, a carbon dioxide, water, other resultants, etc. are usually included besides the maleic anhydride. Usually recovery of the maleic anhydride from the reaction generation



gas which flows out of a reactor contacts reaction generation gas, an organic solvent, or an aqueous solvent, carries out uptake of the maleic anhydride into this solvent, and is performed by collecting maleic anhydrides from this solvent.

[0021] When manufacturing acrylonitrile, nitrogen, a carbon dioxide, etc. are usually used for a raw material as a hydrocarbon as the air to which ammonia was carried out as an ammoxidation raw material, and enrichment of air and the oxygen was carried out as oxygen content gas for the hydrocarbons of the carbon number 3 of a propane, a propylene, etc. again and oxygen or those mixed gas, and inert gas.

[0022] As a catalyst, a V-Sb system oxide catalyst, a V-Sb-W system oxide catalyst, a V-Sb-Sn-Cu-Bi system oxide catalyst, a V-Sb-Sn-Cu-Te system oxide catalyst, a Sb-Sn system oxide catalyst, an As-Sn system oxide catalyst, a Mo-Sn system oxide catalyst, a V-Cr system oxide catalyst, a Mo-Bi-Fe-aluminum system oxide catalyst, a Mo-Cr-Te system oxide catalyst, Although a Mo-Bi-Cr system oxide catalyst, a Cr-Sb-W system oxide catalyst, a Mo-Sb-W system oxide catalyst, a Mo-Bi-Cr-Nb system oxide catalyst, a Mo-V-Te system oxide catalyst, a Mo-V-Sb system oxide catalyst, etc. can be used. The compound metal oxide catalyst which uses one [ at least ] element of molybdenum, vanadium and a tellurium, or the antimony as an indispensable component especially is used. For example, molybdenum, vanadium, X and Y, and oxygen (at least one sort of X of a tellurium and the antimony) Y Niobium, a tantalum, a tungsten, titanium, aluminum, a zirconium, Chromium, manganese, iron, a ruthenium, cobalt, a rhodium, nickel, one or more sorts of elements chosen from the group which consists of palladium, platinum, a bismuth, boron, an indium, Lanthanum, germanium, rare earth elements, alkali metal, and alkaline earth metal -- being shown -- the compound metal oxide catalyst used as an indispensable component is desirable. What the abundance of these catalyst configuration element is especially expressed with following type  $0.25 < r_{Mo} < 0.980.003 < r_V < 0.50.003 < r_X < 0.50 < r_Y < 0.5$  (however,  $r_{Mo}$ ,  $r_V$ ,  $r_X$ , and  $r_Y$  express the mole fraction of Mo, V, X, and Y to the sum total of the above-mentioned indispensable component except oxygen) to is desirable.

[0023] Such a catalyst can be manufactured by the approach indicated by JP,2-257,A, JP,5-148212,A, JP,5-208136,A, JP,6-279351,A, JP,9-157241,A, etc. Although a reaction can use a fluid bed reactor or a fixed bed reactor, it is desirable to use the fluid bed reactor which control of reaction temperature tends to carry out. Reaction temperature is usually 200-500 degrees C. Although a reaction can be performed also under reduced pressure or pressurization, it is usually carried out in the range of 0.2MPaG(s) from ordinary pressure.

[0024] In the reactant gas which flows out of a reactor, the resultant of unreacted oxygen, coal-for-coke-making-ized hydrogen, ammonia, the acrylic acid that carries out a byproduction to a list, an olefin acid, a carbon monoxide, a carbon dioxide, and others etc. is usually included besides acrylonitrile. Usually, separation of the acrylic acid from reaction generation gas washes reaction generation gas in a sulfuric-acid water solution, removes unreacted ammonia, and is performed by collecting acrylonitrile and acrylic acids as main liquid by subsequently washing with water.

[0025]

[Example] Hereafter, although an example explains this invention to a detail further, this invention is not limited by the following examples unless the summary is exceeded. Drawing 2 shows the vapor-phase-oxidation reaction structure of a system by one example of this invention. The element shown with reference numbers 1-9 in drawing 2 is equivalent to the element of the same reference number in drawing 1. Moreover, although the element shown in drawing 2 as a control unit 10 expresses the control unit of the hardware which has each function of the 1st control means 8 and the 2nd control means 9, it does not necessarily need to be single equipment. The main contents of processing are shown to the interior of the 1st control means 8 and the 2nd control means 9 by the flow. What kind of PID-control equipment or feedback control equipment is sufficient as a control device 10, and it calculates and outputs a manipulated variable (MV) by considering a control variable (valve flow coefficient) as an input.

[0026] The main configurations and the actuation of a system which are shown in drawing 2 are the same as that of the system of drawing 1, and serve as one reactor 1, and one or decollator 2-A beyond it - 2-N from the recycle loop formation 3, and a part for new supply of a hydrocarbon, oxygen, air or inert gas, and other gaseous-phase raw materials is supplied to a reactor 1. The resultant from a reactor 1, a by-product, and an unnecessary component are taken out by the decollator, or are removed. There is outflow from a reactor 1 in R points, there is outflow from decollator 2-A in an A point, and there is outflow from decollator 2-N in N point like the following. The outflow of the last N point is recycled by the reactor 1 as a part of feeder current.

[0027] In the 1st control means 8, the sum total flow rate of a reactor inlet port or the flow rate of the outflow of any one or more points of R points - the N point can be taken as a control variable (valve flow coefficient). Flow rates should just be flow rates usually used, such as a mass flow rate, a molar flow rate, and a volumetric flow rate. In the case of the example of illustration, the 1st control means 8 detects the flow rate of the outflow from a reactor 1 by R points, compares the detection result with the desired value of the amount of outflow set up beforehand, and calculates the air according to the amount of deflection which detected and detected deflection, or the actuation value over the supply flow

rate of inert gas. The actuation value of the result of an operation is fed back to the flow rate directions regulator 6 as a manipulated variable (MV). In the flow rate directions regulator 6, the flow rate F3 of the air supplied to a reactor 1 or inert gas is adjusted based on the value of the inputted manipulated variable (MV).

[0028] Processing of the 2nd control means 9 consists of two of the processings which calculate the actuation value of an oxygen flow rate required to detect the flow rate of the processing which detects the oxygen density in a system and asks for the flow rate of a target hydrocarbon and oxygen, the hydrocarbon of feed gas, and oxygen, and adjust the flow rate to desired value.

[0029] In the 1st processing, the flow rate set point of a hydrocarbon and oxygen which detects the oxygen density in the outflow of any one or more points of R points - the N point, and sets it as an actuation target as a control variable (valve flow coefficient) based on the detected oxygen density and the oxygen density desired value set up beforehand is calculated. In the case of the example of illustration, the oxygen density of the outflow from a reactor 1 is detected by R points, the acquired oxygen density detection value is compared with the oxygen density desired value set up beforehand, deflection is detected, the desired value of the flow rate of the hydrocarbon according to the amount of deflection and oxygen is calculated, and it is considering as the set point for the next processing.

[0030] In the 2nd processing, the hydrocarbon supply flow rate and oxygen supply flow rate to a reactor 1 are detected, the flow rate set point of the value of the flow rate of a hydrocarbon and oxygen, a target hydrocarbon, and oxygen which asked for and asked for the flow rate of a hydrocarbon and oxygen is compared, deflection is detected, the oxygen flow rate actuation value of the manipulated variable (MV) according to the amount of deflection is calculated, and delivery and modification are directed to the flow rate directions regulator 5. In the flow rate directions regulator 5, the oxygen supply flow rate F2 to a reactor 1 is changed based on the directed actuation value.

[0031]

[Effect of the Invention] According to this invention, it becomes possible by adjusting directly or indirectly the sum total flow rate of a reactor inlet port to maintain the supply flow rate to each decollator, and the amount of outflow from each decollator at a request value. Furthermore, it becomes possible by adjusting directly or indirectly the ratio of the oxygen density of a reactor outlet, and hydrocarbon concentration to keep the gas presentation of a reactor inlet port consequent to a request value. While it becomes easy to stabilize the reaction in a reactor by this and a reaction invert ratio and major product selectivity are stabilized, stable operation of the whole vapor-phase-oxidation reaction system equipped with the recycle loop formation also of stabilization of operation of the separation recovery system of a product or a resultant is attained by becoming easy.

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**PRIOR ART**

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[Description of the Prior Art] Under existence of a catalyst, as a vapor-phase-oxidation reaction to which a hydrocarbon and oxygen content gas are made to react, there is the catalytic-oxidation approach of a hydrocarbon, for example, and manufacture of the maleic anhydride by oxidation of the hydrocarbon of a carbon number 4, manufacture of the acrylonitrile by the ammoxidation of the hydrocarbon of a carbon number 3, manufacture of the ethylene dichloride by the oxychlorination of ethylene, etc. are known widely. In recent years, in order to produce the target product efficiently in these vapor-phase-oxidation reactions, while raising the selectivity to a product by stopping the invert ratio of the hydrocarbon in a reactor low, the recycling law through which collects unreacted hydrocarbons and a reactor is made to circulate is proposed.

[0003] However, although operation was able to be continued to stability in the conventional process by controlling a reaction condition (the flow rate of each gas, and temperature, a pressure, the amount of catalysts) independently, respectively when reaction results changed, for example In the case of a recycle process, in order that the gas which came out of the reactor may finally return to a reactor via a decollator etc. again For example, when reaction results change, even if it was going to perform a reaction conditional control like before, in order that the flow rate of recycle gas and a presentation might change influenced by the reaction condition of modification, if it remained as it is, it was difficult [ it ] to fall and wear in the regular condition, and for there to be nothing and to continue oxidation reaction to stability as a result with Lycium chinense.

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[Translation done.]

JAPANESE

[JP,2001-114706,A]

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CLAIMS DETAILED DESCRIPTION TECHNICAL FIELD PRIOR ART EFFECT OF THE INVENTION  
TECHNICAL PROBLEM MEANS EXAMPLE DESCRIPTION OF DRAWINGS DRAWINGS

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TECHNICAL PROBLEM

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[Problem(s) to be Solved by the Invention] This invention aims the vapor-phase-oxidation reaction system of the hydrocarbon which has the recycle process through which collects unreacted hydrocarbons and a reactor is made to circulate at efficient and offering the control approach for operating to stability, and a control unit.

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MEANS

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[Means for Solving the Problem] At least one reactor with which the raw material with which this invention contains a hydrocarbon, oxygen and air, or inert gas is supplied, In the vapor-phase-oxidation reaction system equipped with the recycle loop formation which returns one combined with this reactor or the decollator beyond it, and the outflow from any one or more decollators to a reactor as a part of feeder current By establishing in parallel the 1st control system which controls directly or indirectly the sum total flow rate of a reactor inlet port, and the 2nd control system which controls the distributed gas presentation to a reactor, or the exhaust-gas presentation from a reactor to a request value Efficient and stable operation of a system is enabled and it is constituted as follows.

[0006] (1) The vapor-phase-oxidation reaction system by this invention At least one reactor with which the raw material containing a hydrocarbon, oxygen and air, or inert gas is supplied, It has the recycle loop formation which returns one combined with this reactor or the decollator beyond it, and the outflow from any one or more decollators to a reactor as a part of feeder current. The sum total flow rate of a reactor inlet port or the amount of outflow from this reactor, and the 1st control system by which even the above controls one side or the both sides of each stage of a decollator with the amount of outflow from any one or more to a request value, [ beyond it ] It is characterized by the configuration which established in parallel the 2nd control system which controls one side or the both sides of a gas presentation of the presentation of all the feeder current to a reactor, the outlet of a reactor, or the outlet of any one or more decollators to a request value.

[0007] (2) The control approach of the vapor-phase-oxidation reaction system by this invention At least one reactor with which the raw material containing a hydrocarbon, oxygen and air, or inert gas is supplied, In the vapor-phase-oxidation reaction system equipped with the recycle loop formation which returns one combined with this reactor or the decollator beyond it, and the outflow from any one or more decollators to a reactor as a part of feeder current As the 1st control, the sum total flow rate or the amount of outflow from this reactor of a reactor inlet port, Even the above or one side or the both sides from any one or more of each stage of a decollator with the amount of outflow beyond it It controls to a request value by changing the flow rate of the air supplied to a vapor-phase-oxidation reaction system, or inert gas. Further as the 2nd control Even the above the oxygen density in one side or the both sides of each stage of a decollator with outflow from either with the outflow from a reactor [ beyond it ] It is characterized by the configuration controlled to a request value by changing the flow rate of the hydrocarbon supplied to a vapor-phase-oxidation reaction system, or the flow rate of oxygen based on the flow rate of the hydrocarbon supplied to a vapor-phase-oxidation reaction system, and oxygen.

[0008] (3) The control unit of the vapor-phase-oxidation reaction system by this invention At least one reactor with which the raw material containing a hydrocarbon, oxygen and air, or inert gas is supplied, It has the recycle loop formation which returns one combined with this reactor or the decollator beyond it, and the outflow from any one or more decollators to a reactor as a part of feeder current. The sum total flow rate of a reactor inlet port or the amount of outflow from this reactor, and even the above or one side or the both sides from any one or more of each stage of a decollator with the amount of outflow beyond it The 1st control means controlled to a request value by changing the flow rate of the air supplied to a vapor-phase-oxidation reaction system, or inert gas, Even the above the oxygen density in one side or the both sides of each stage of a decollator with outflow from either with the outflow from a reactor [ beyond it ] It is characterized by the configuration which has the 2nd control means controlled to a request value by changing the flow rate of the hydrocarbon supplied to a vapor-phase-oxidation reaction system, or the flow rate of oxygen based on the flow rate of the hydrocarbon supplied to a vapor-phase-oxidation reaction system, and oxygen.

[0009] The outline configuration of the vapor-phase-oxidation reaction system which applied this invention is shown in drawing 1 by the instantiation-approach. Hereafter, the detail of this invention is explained based on drawing 1 . The vapor-phase-oxidation reaction system of drawing 1 has at least one reactor 1, one, or decollator 2-A beyond it - 2-N,

and recycle loop formation 3. A part for new supply of the hydrocarbon supplied to a reactor 1, oxygen, air or inert gas, and other gaseous-phase raw materials is adjusted to a flow rate F1, F2, F3, and F4 by the flow rate directions regulators 4, 5, 6, and 7, respectively. Moreover, although the illustration abbreviation is carried out, the catalyst is made to exist in a reactor 1.

[0010] The resultant from a reactor 1, a by-product, or an unnecessary component is taken out from each of decollator 2-A - 2-N by arbitration, or is removed. R points show the outflow from a reactor 1, and, as for an A point, the outflow from first decollator 2-A, --, N point show the outflow from Nth decollator 2-N. The outflow from last or Nth decollator 2-N is recycled by the reactor 1 through the recycle loop formation 3 as a part of feeder current. This recycle flow rate is expressed with F5. Thereby, the sum total flow rate of the inlet port of a reactor 1 is given by  $(F1+F2+F3+F4+F5)$ . Moreover, the presentation of all the feeder current to a reactor 1 is given with the sum total flow rate  $(F1+F2+F3+F4+F5)$  of each component flow rate / reactor inlet port of a reactor inlet port.

[0011] Furthermore by this invention, at least two independent control systems, the 1st and the 2nd, are established respectively through the 1st control means 8 and 2nd control means 9. The 1st control system using the 1st control means 8 The sum total flow rate or the amount of outflow from this reactor of the inlet port of a reactor 1, It is for even the above to control one side or the both sides of each stage of a decollator with the amount of outflow from any one or more to a request value. In the example of illustration [ beyond it ] The flow rate of the outflow from a reactor 1 was detected by R points, and the flow rate F3 of the air supplied to a reactor 1 or inert gas is changed so that the detected flow rate may be in agreement with desired value. The 2nd control system Moreover, the presentation of all the feeder current to a reactor 1 or the outlet of a reactor 1, It is for controlling the gas presentation in the outlet of one or more things of decollator 2-A - 2-N to a request value. Or in the example of illustration The gas presentation of the outflow from a reactor 1 was detected by R points, and the flow rate F1/F2 of the hydrocarbon supplied to a reactor 1 and oxygen is changed so that the detected gas presentation may be in agreement with the set point. The material gas containing a hydrocarbon, oxygen, air, or inert gas may be directly supplied to a reactor or a decollator, and may be supplied to the feeder current or the outflow to a decollator.

[0012] As the 1st control means 8 and 2nd control means 9, it can use also with what type of PID (proportional-integral-derivative) control device also with what kind of a feedback mold control device or what kind of kind of feedforward mold control device. In the example shown in drawing 2, a control system including the 1st control means 8 controls the flow rate directions regulator 6 to lower the air in a reactor inlet port, or the supply flow rate F3 of inert gas, when the amount of outflow measured by R points of for example, a reactor outlet increases to desired value, and it functions as maintaining the amount of outflow in a reactor outlet at a request value.

[0013] moreover, when the 1st control means 8 detects the flow rate of either the A point of each decollator outlet - N point, for example, the outflow of M point, unlike the example of illustration, the flow rate directions regulator 6 is controlled and the flow rate F3 of air or inert gas is changed so that the flow rate of M point may be maintained at a request value. Therefore, when the flow rate of M points decreases to the set point, it controls in the direction to which the flow rate F3 of air or inert gas is made to increase, and it operates so that the flow rate of M points may be maintained at a request value.

[0014] Next, the 2nd control means 9 measures the oxygen density in the outflow of R points, and a control system including the 2nd control means 9 controls it to lower the desired value of the flow rate (for example, it considers as an oxygen flow rate / hydrocarbon flow rate) of the hydrocarbon in a reactor inlet port, and oxygen, when the measured value increases to the desired value of the oxygen density set up beforehand. The control system of the flow rate of the hydrocarbon contained in the 2nd control means and oxygen measures the flow rate (for example, it considers as an oxygen flow rate / hydrocarbon flow rate) of the hydrocarbon of a reactor inlet port, and oxygen, when increasing to desired value, control the flow rate directions regulator 5 of the oxygen in a reactor inlet port, and the flow rate of the hydrocarbon of a reactor inlet port and oxygen is made to follow desired value, and it functions as keeping final the oxygen density in the outflow of R points to the desired value set up beforehand. Or the flow rate of the hydrocarbon of a reactor inlet port and oxygen is measured, when increasing to desired value, the flow rate directions regulator 4 of the hydrocarbon in a reactor inlet port may be controlled, the flow rate of the hydrocarbon of a reactor inlet port and oxygen may be made to follow desired value unlike the case of illustration, and, finally the oxygen density in the outflow of R points may be operated with maintaining at the desired value set up beforehand. In addition, when detecting and controlling the oxygen density in outflow about R points - two or more N points unlike the case of illustration, a control variable (valve flow coefficient) is constituted using the weighted average of each detection value, and other suitable performance indices, and it is made to maintain at desired value by the same control technique.

[0015] The exhaust-gas flow rate or recycle gas flow rate from each distributed gas flow rate, reactor, or decollator to a reaction system is measured by quantity-of-gas-flow measurement means usually used, such as a differential pressure

type flowmeter, an eddy type flowmeter, a coriolis type flowmeter, and a variable area flowmeter. The oxygen density in the outflow from a reactor or a decollator is measured by oxygen density measurement means usually used, such as an analyzer which can measure various online oxygen analyzers and an oxygen density, and hydrocarbon concentration is also measured by various online analyzers, such as an infrared analyzer and a mass spectrometer. In addition, it is desirable to use the means which can measure [ rather than ] concentration continuously in this case using a discontinuous analysis means like a gas chromatograph.

[0016] A decollator may separate the resultant, by-product, or unnecessary component from a reactor or a decollator, and may divide the outflow from a reactor or a decollator into two or more flow. As a decollator, well-known decollators, such as an absorption decollator, a condensation decollator, adsorption separation equipment, and a distillation decollator, can usually be used.

[0017] This invention is applicable to manufacture of the acrylonitrile by the ammoxidation of the hydrocarbon of the carbon number 3 of manufacture of the maleic anhydride by oxidation of the hydrocarbon of the carbon number 4 of the vapor-phase-oxidation reaction to which a hydrocarbon and oxygen content gas are made to react, for example, butane, a butene, a butadiene, etc., a propane, a propylene, etc., and manufacture of the ethylene dichloride by the oxychlorination of ethylene under existence of a catalyst.

[0018] When manufacturing a maleic anhydride, nitrogen, a carbon dioxide, etc. are usually used for a raw material as the air to which the hydrocarbons of the carbon number 4 of butane, a butene, a butadiene, etc. were carried out as a hydrocarbon, and enrichment of air and the oxygen was carried out as oxygen content gas and oxygen or those mixed gas, and inert gas. Especially the thing that makes an active ingredient the multiple oxide (vanadium-Lynn system multiple oxide) which uses vanadium and Lynn as a main configuration element as a catalyst, and makes vanadic pyrophosphate an active ingredient especially is desirable.

[0019] Such a catalyst can be manufactured by the approach indicated by Chem.Rev.88, 55-80 pages (1988), JP,59-95933,A, the U.S. Pat. No. 4,472,527 specification, the U.S. Pat. No. 4,520,127 specification, etc. A reaction can use a fluid bed reactor or a fixed bed reactor, and is made to usually react at 300-600 degrees C.

[0020] In the reaction generation gas which flows out of a reactor, unreacted oxygen and coal-for-coke-making-ized hydrogen, the carbon monoxide which carries out a byproduction to a list, a carbon dioxide, water, other resultants, etc. are usually included besides the maleic anhydride. Usually recovery of the maleic anhydride from the reaction generation gas which flows out of a reactor contacts reaction generation gas, an organic solvent, or an aqueous solvent, carries out uptake of the maleic anhydride into this solvent, and is performed by collecting maleic anhydrides from this solvent.

[0021] When manufacturing acrylonitrile, nitrogen, a carbon dioxide, etc. are usually used for a raw material as a hydrocarbon as the air to which ammonia was carried out as an ammoxidation raw material, and enrichment of air and the oxygen was carried out as oxygen content gas for the hydrocarbons of the carbon number 3 of a propane, a propylene, etc. again and oxygen or those mixed gas, and inert gas.

[0022] As a catalyst, a V-Sb system oxide catalyst, a V-Sb-W system oxide catalyst, A V-Sb-Sn-Cu-Bi system oxide catalyst, a V-Sb-Sn-Cu-Te system oxide catalyst, A Sb-Sn system oxide catalyst, an As-Sn system oxide catalyst, a Mo-Sn system oxide catalyst, A V-Cr system oxide catalyst, a Mo-Bi-Fe-aluminum system oxide catalyst, a Mo-Cr-Te system oxide catalyst, Although a Mo-Bi-Cr system oxide catalyst, a Cr-Sb-W system oxide catalyst, a Mo-Sb-W system oxide catalyst, a Mo-Bi-Cr-Nb system oxide catalyst, a Mo-V-Te system oxide catalyst, a Mo-V-Sb system oxide catalyst, etc. can be used The compound metal oxide catalyst which uses one [ at least ] element of molybdenum, vanadium and a tellurium, or the antimony as an indispensable component especially is used. For example, molybdenum, vanadium, X and Y, and oxygen (at least one sort of X of a tellurium and the antimony) Y Niobium, a tantalum, a tungsten, titanium, aluminum, a zirconium, Chromium, manganese, iron, a ruthenium, cobalt, a rhodium, nickel, one or more sorts of elements chosen from the group which consists of palladium, platinum, a bismuth, boron, an indium, Lynn, germanium, rare earth elements, alkali metal, and alkaline earth metal -- being shown -- the compound metal oxide catalyst used as an indispensable component is desirable. What the abundance of these catalyst configuration element is especially expressed with following type  $0.25 < r_{Mo} < 0.980.003 < r_V < 0.50.003 < r_X < 0.50 < r_Y < 0.5$  (however,  $r_{Mo}$ ,  $r_V$ ,  $r_X$ , and  $r_Y$  express the mole fraction of Mo, V, X, and Y to the sum total of the above-mentioned indispensable component except oxygen) to is desirable.

[0023] Such a catalyst can be manufactured by the approach indicated by JP,2-257,A, JP,5-148212,A, JP,5-208136,A, JP,6-279351,A, JP,9-157241,A, etc. Although a reaction can use a fluid bed reactor or a fixed bed reactor, it is desirable to use the fluid bed reactor which control of reaction temperature tends to carry out. Reaction temperature is usually 200-500 degrees C. Although a reaction can be performed also under reduced pressure or pressurization, it is usually carried out in the range of 0.2MPaG(s) from ordinary pressure.

[0024] In the reactant gas which flows out of a reactor, the resultant of unreacted oxygen, coal-for-coke-making-ized

hydrogen, ammonia, the acrylic acid that carries out a byproduction to a list, an olefin acid, a carbon monoxide, a carbon dioxide, and others etc. is usually included besides acrylonitrile. Usually, separation of the acrylic acid from reaction generation gas washes reaction generation gas in a sulfuric-acid water solution, removes unreacted ammonia, and is performed by collecting acrylonitrile and acrylic acids as main liquid by subsequently washing with water.

[0025]

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[Translation done.]



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EXAMPLE

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[Example] Hereafter, although an example explains this invention to a detail further, this invention is not limited by the following examples unless the summary is exceeded. Drawing 2 shows the vapor-phase-oxidation reaction structure of a system by one example of this invention. The element shown with reference numbers 1-9 in drawing 2 is equivalent to the element of the same reference number in drawing 1. Moreover, although the element shown in drawing 2 as a control unit 10 expresses the control unit of the hardware which has each function of the 1st control means 8 and the 2nd control means 9, it does not necessarily need to be single equipment. The main contents of processing are shown to the interior of the 1st control means 8 and the 2nd control means 9 by the flow. What kind of PID-control equipment or feedback control equipment is sufficient as a control device 10, and it calculates and outputs a manipulated variable (MV) by considering a control variable (valve flow coefficient) as an input.

[0026] The main configurations and the actuation of a system which are shown in drawing 2 are the same as that of the system of drawing 1, and serve as one reactor 1, and one or decollator 2-A beyond it - 2-N from the recycle loop formation 3, and a part for new supply of a hydrocarbon, oxygen, air or inert gas, and other gaseous-phase raw materials is supplied to a reactor 1. The resultant from a reactor 1, a by-product, and an unnecessary component are taken out by the decollator, or are removed. There is outflow from a reactor 1 in R points, there is outflow from decollator 2-A in an A point, and there is outflow from decollator 2-N in N point like the following. The outflow of the last N point is recycled by the reactor 1 as a part of feeder current.

[0027] In the 1st control means 8, the sum total flow rate of a reactor inlet port or the flow rate of the outflow of any one or more points of R points - the N point can be taken as a control variable (valve flow coefficient). Flow rates should just be flow rates usually used, such as a mass flow rate, a molar flow rate, and a volumetric flow rate. In the case of the example of illustration, the 1st control means 8 detects the flow rate of the outflow from a reactor 1 by R points, compares the detection result with the desired value of the amount of outflow set up beforehand, and calculates the air according to the amount of deflection which detected and detected deflection, or the actuation value over the supply flow rate of inert gas. The actuation value of the result of an operation is fed back to the flow rate directions regulator 6 as a manipulated variable (MV). In the flow rate directions regulator 6, the flow rate F3 of the air supplied to a reactor 1 or inert gas is adjusted based on the value of the inputted manipulated variable (MV).

[0028] Processing of the 2nd control means 9 consists of two of the processings which calculate the actuation value of an oxygen flow rate required to detect the flow rate of the processing which detects the oxygen density in a system and asks for the flow rate of a target hydrocarbon and oxygen, the hydrocarbon of feed gas, and oxygen, and adjust the flow rate to desired value.

[0029] In the 1st processing, the flow rate set point of a hydrocarbon and oxygen which detects the oxygen density in the outflow of any one or more points of R points - the N point, and sets it as an actuation target as a control variable (valve flow coefficient) based on the detected oxygen density and the oxygen density desired value set up beforehand is calculated. In the case of the example of illustration, the oxygen density of the outflow from a reactor 1 is detected by R points, the acquired oxygen density detection value is compared with the oxygen density desired value set up beforehand, deflection is detected, the desired value of the flow rate of the hydrocarbon according to the amount of deflection and oxygen is calculated, and it is considering as the set point for the next processing.

[0030] In the 2nd processing, the hydrocarbon supply flow rate and oxygen supply flow rate to a reactor 1 are detected, the flow rate set point of the value of the flow rate of a hydrocarbon and oxygen, a target hydrocarbon, and oxygen which asked for and asked for the flow rate of a hydrocarbon and oxygen is compared, deflection is detected, the oxygen flow rate actuation value of the manipulated variable (MV) according to the amount of deflection is calculated, and delivery and modification are directed to the flow rate directions regulator 5. In the flow rate directions regulator 5, the oxygen supply flow rate F2 to a reactor 1 is changed based on the directed actuation value.

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DESCRIPTION OF DRAWINGS

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[Brief Description of the Drawings]

[Drawing 1] It is the basic block diagram of the vapor-phase-oxidation reaction system by this invention.

[Drawing 2] It is 1 example block diagram of this invention vapor-phase-oxidation reaction system.

[Description of Notations]

- 1 Reactor
- 2-A - 2-N Decollator
- 3 Recycle Loop Formation
- 4-7 Flow rate directions regulator
- 8 1st Control Means
- 9 2nd Control Means
- 10 Control Unit

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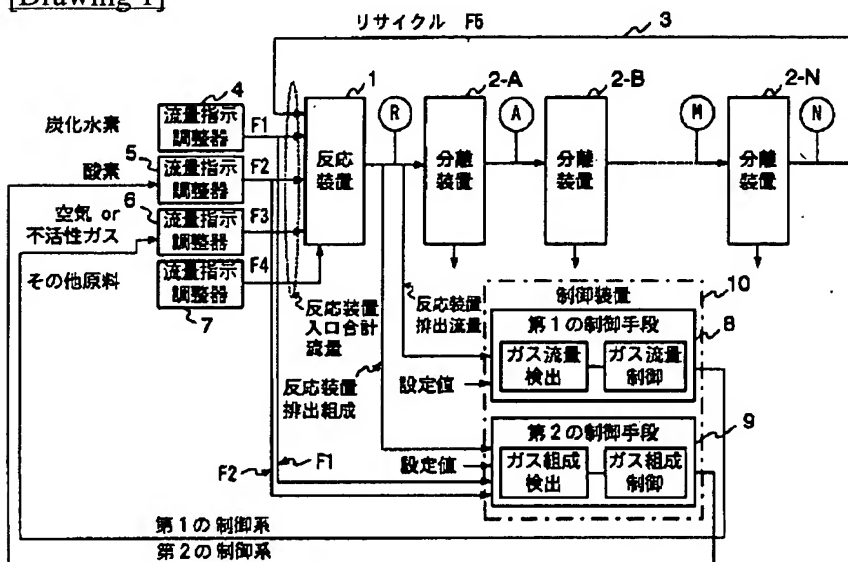
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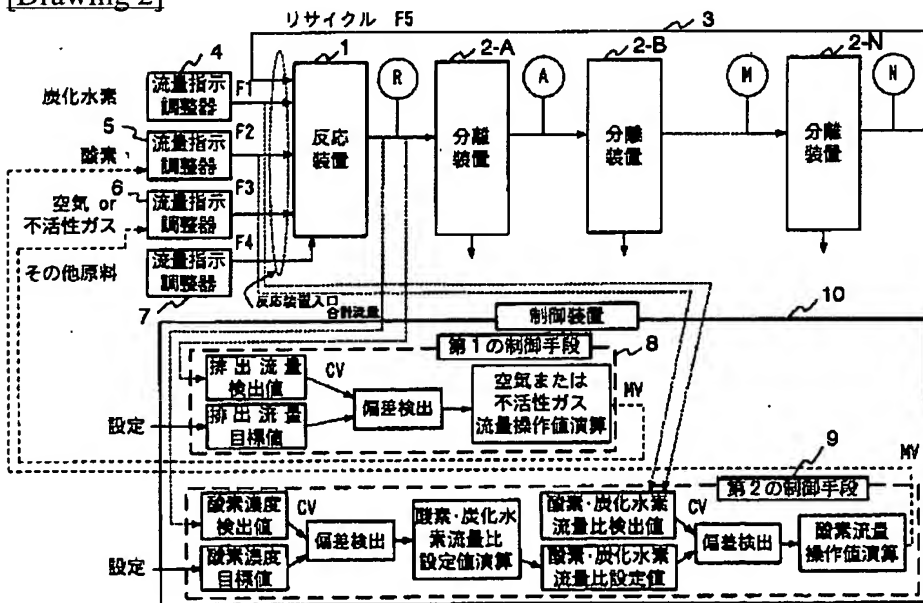
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## DRAWINGS

[Drawing 1]



[Drawing 2]



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